WANL-PR(P)-004
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DETERMINATION OF THE WELDABILITY AND ELEVATED TEMPERATURE STABILITY OF REFRACTORY METAL ALLOYS

Fourth Quarterly Report

by

G. G. Lessmann and D. R. Stoner

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LEWIS RESEARCH CENTER
UNDER CONTRACT NAS3-2540

Astronuclear Laboratory
Westinghouse Electric Corporation

Astronuclear

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FOURTH QUARTERLY REPORT

Covering the Period

March 21, 1964 to June 21, 1964

Prepared For

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
Contract NAS 3-2540

Technical Management
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FOREWORD

This report describes work accomplished under Contract NAS 3-2540 during the period March 21, 1964 to June 21, 1964. This program is being administered by R. T. Begley of the Astronuclear Laboratory, Westinghouse Electric Corporation. G. G. Lessmann and D. R. Stoner performed the experimental investigations.

P. E. Moorhead of the National Aeronautics and Space Administration, is Technical Manager of this program.

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I. INTRODUCTION

This is the Fourth Quarterly Progress Report describing work accomplished under Contract NAS 3-2540. The objective of this program is to determine the weldability and long time elevated temperature stability of promising refractory metal alloys in order to determine those most suitable for use in advanced alkali-metal space electric power systems. A detailed discussion of the program and program objectives was presented in the First Quarterly Report. Alloys included in this investigation are listed in Table 1.

Process and test controls employed throughout this program emphasize the important influence of interstitial elements on the properties of refractory metal alloys. Stringent process and test procedures are required, including continuous monitoring of the TIG weld chamber atmosphere, electron beam welding at low pressures, aging in furnaces employing hydrocarbon free pumping systems providing pressures less than 10^{-8} Torr, and chemical sampling following successive stages of the evaluation for verification of these process controls.

Equipment requirements and set-up, and procedures for welding and testing, have been described in previous progress reports. Any improvements in processes, changes in procedures, or additional processes and procedures are described in this report.

TABLE 1 - Alloys Included in the Weldability and Thermal Stability Evaluations

Alloy	Nominal Composition Weight Percent
AS-55	Cb-5W-1Zr-0.2Y-0.06C
B-66	Cb-5Mo-5V-1Zr
C-129Y	Cb-10W-10Hf+Y
Cb-752	Cb-10W-2.5Zr
D-43	Cb-10W-1Zr-0.1C
FS-85	Cb-27Ta-10W-1Zr
SCb-291	Cb-10W-10Ta
T-111	Ta-8W-2Hf
T-222	Ta-9.6W-2.4Hf-0.01C
Ta-10W	Ta-10W
W-25Re	W-25Re
W	Unalloyed
Sylvania "A"	W-0.5Hf-0.02C

Note: All alloys to be from arc-cast and/or electron beam melted material except Sylvania "A"

II. SUMMARY

The delivery status of the thirteen alloys included in this program is shown in Table 2. Orders for nine of these have been filled.

TIG sheet butt welds for the weld parameter study have been completed for all of the available alloys except W-25Re and unalloyed tungsten. Weld and bend test data on the first series of these welds was reported in the Third Quarterly Report. Testing of the remaining welds is in process and will be reported in the Fifth Quarterly Report. A summary of current bend test results is given in Figure 1.

EB welds for the weld parameter study have been completed for sheet material of four alloys and welding was initiated for two others. Those completed and tested include FS-85 and Ta-10W (reported in the Third Quarterly) while results for Cb-752 and SCb-291 are included in this report. W-25Re and D-43 are presently being welded and tested. Twelve welds are required for each parameter test series per alloy per welding process (TIG or EB). Based on test results of the first four alloys, the parameter selection for the EB welding was modified to more clearly discriminate between parameter variation effects on weld bend transition temperature. The purpose of these changes was to more completely exploit the wide range of transition temperature obtainable, thereby permitting a more comprehensive comparison of the alloys to be made.

All available plate material has been machined with the double "U" weld joint configuration in preparation for welding and testing. The adequacy of this joint design and welding qualification for each alloy using this design was demonstrated during the previous quarter.

The bead-on-plate restraint patch test was made for the W-25Re alloy. A tendency toward loss of ductility in the heat affected zone and weld, and consequent cracking during welding was demonstrated.

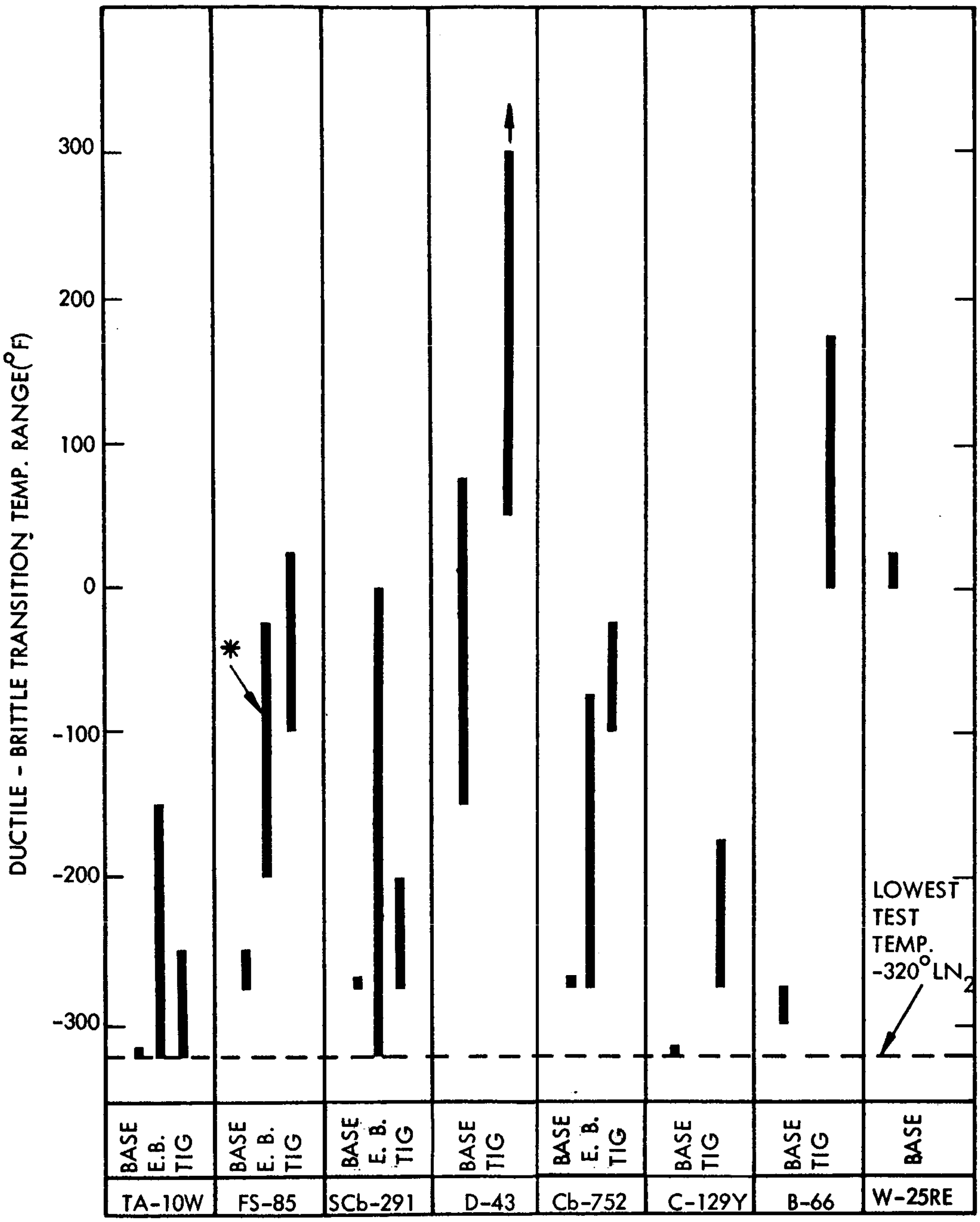
The copper clamp down inserts in the TIG sheet butt welding fixture were replaced with molybdenum. Improved welding was realized at the narrow clamp spacings at which arc edge flashing had occurred using copper clamp bars. A special backup insert was also designed and fabricated. This insert is designed for weld preheating and equipped with an integral heater. It will be required for welding unalloyed tungsten, Sylvania "A" and perhaps W-25Re.

In a continuing effort to systematically improve the TIG welding atmosphere, a 600 watt titanium getter ribbon was tested, latex natural rubber gloves were purchased for evaluation, and a high purity gas supply is being evaluated.

TABLE 2 - Alloy Procurement and Delivery Schedule

Alloy	Approval	Quotation	Ordered	Shipping Date	Actual Delivery			Supplier
					Sheet	Plate	Wire	
AS-55	8/12/63	10/1/63	1/29/64	5/1/64		—[3]	—[3]	Gen. Electric (Cleveland)
B-66	8/12/63	8/19/63	8/29/63	10/18/63	3/3/64	3/3/64	11/8/63	Westinghouse
C-129Y	8/12/63	9/20/63	10/2/63	11/30/63	12/24/63	3/13/64	3/13/64	Wah Chang
Cb-752	8/12/63	9/19/63	10/21/63	11/30/63	12/31/63	12/18/63	12/31/63	Haynes
D-43	8/12/63	8/17/63	9/3/63	11/8/63	11/15/63	10/18/63	2/12/64	Du Pont
FS-85	8/12/63	8/12/63	8/22/63	1/30/64	3/6/64	1/6/64	3/7/64	Fansteel[1]
SCb-291	8/12/63	9/17/63	10/2/63	1/30/64	1/9/64	1/8/64	12/6/63	Fansteel
T-111	8/12/63	8/16/63 6/25/64[2] 6/25/64	9/27/63 8/5/64 —[5]	10/28/63 9/28/64 —[5]	—[4] —[4]	12/31/63 —[4] —[4]	—[4] —[4] 8/14/64	NRC Wah Chang Westinghouse
T-222	2/28/64	6/29/64[2]	7/20/64	9/28/64				Wah Chang
Ta-10W	8/12/63	8/12/63	8/22/63	9/30/63	10/21/63	10/3/63	10/17/63	Fansteel
W-25Re	8/12/63	11/26/63	2/1/64	4/1/64	5/29/64	—[3]	—[3]	Wah Chang
W	2/28/64	2/19/64	4/16/64	6/15/64	7/30/64	—[3]	—[3]	Universal Cyclops
Sylvania "A"	6/24/64	5/15/64	5/15/64	9/30/64		—[3]	—[3]	Sylvania

1. Sheet material produced by Fansteel under Contract NOw-63-0231-c and furnished to this program as transferred government owned material.
2. Second procurement action for this material
3. Not included in program.
4. T-111 order split between three suppliers.
5. Converted at Westinghouse Astronuclear Laboratory.



* ALL BENDS 1t RADIUS EXCEPT FS-85 E. B. WELDS WHICH ARE 2t RADIUS

FIGURE 1 - Summary of Current Bend Test Results

III. TECHNICAL PROGRAM

A. ALLOY PROCUREMENT

The procurement status of the alloys included in this program is shown in Table 2. Difficulties encountered in procuring T-111 and T-222 resulted in reordering of these alloys during the past period. The only additional delivery has been unalloyed arc cast tungsten sheet. The as-received microstructure of W and W-25Re sheet are shown in Figure 2. Porosity was noted in the microstructure of one of the W sheets. The hardness of the W is 501 DPH for heat KC1353 and 533 DPH for heat KC1350. The W-25Re hardness is 526 DPH and its 1t bend transition temperature was between 0 and 25°F. (See Figure 3) Hardness, metallography, and bend test data for the other alloys were reported previously.

B. WELDING EVALUATIONS

1. TIG Welding

Sheet welding was completed on seven alloys for the parameter optimization phase. Bend data for the first four welds of each alloy were reported during the last period. The final eight welds of each alloy are presently being tested. This data will be included in the next quarterly report.

In selecting weld parameters for this phase of the evaluation, the effects of variation in weld freezing rate, cooling rate, and unit weld length heat input have been emphasized rather than current, speed and voltage per se. The welding set-up was described previously. A typical welding schedule is shown in Figure 4. The twelve selected welding conditions are indicated in this figure. Weld freezing and cooling rates are closely associated with weld speed and clamp spacing; hence, these were chosen as variables. The unit weld length heat input is related to weld size as well as weld speed. Hence, two different weld target sizes were chosen to identify total heat input effects. Welding currents were then selected to obtain the desired weld size. Target weld sizes were standardized for all alloys. The cross-sectional weld areas selected were 0.0039 and 0.0065 square inches respectively for the small and large welds. These represent welds having average widths of 0.11 inches and 0.18 inches. Standardization of weld sizes represent a practical method for comparing alloys since, typically, any one particular application requires a specific fixed weld size regardless of the alloy selected. (Assuming alloys of approximately the same strengths, hence, same joint size.)

To aid in the selection of weld parameters, curves were developed for

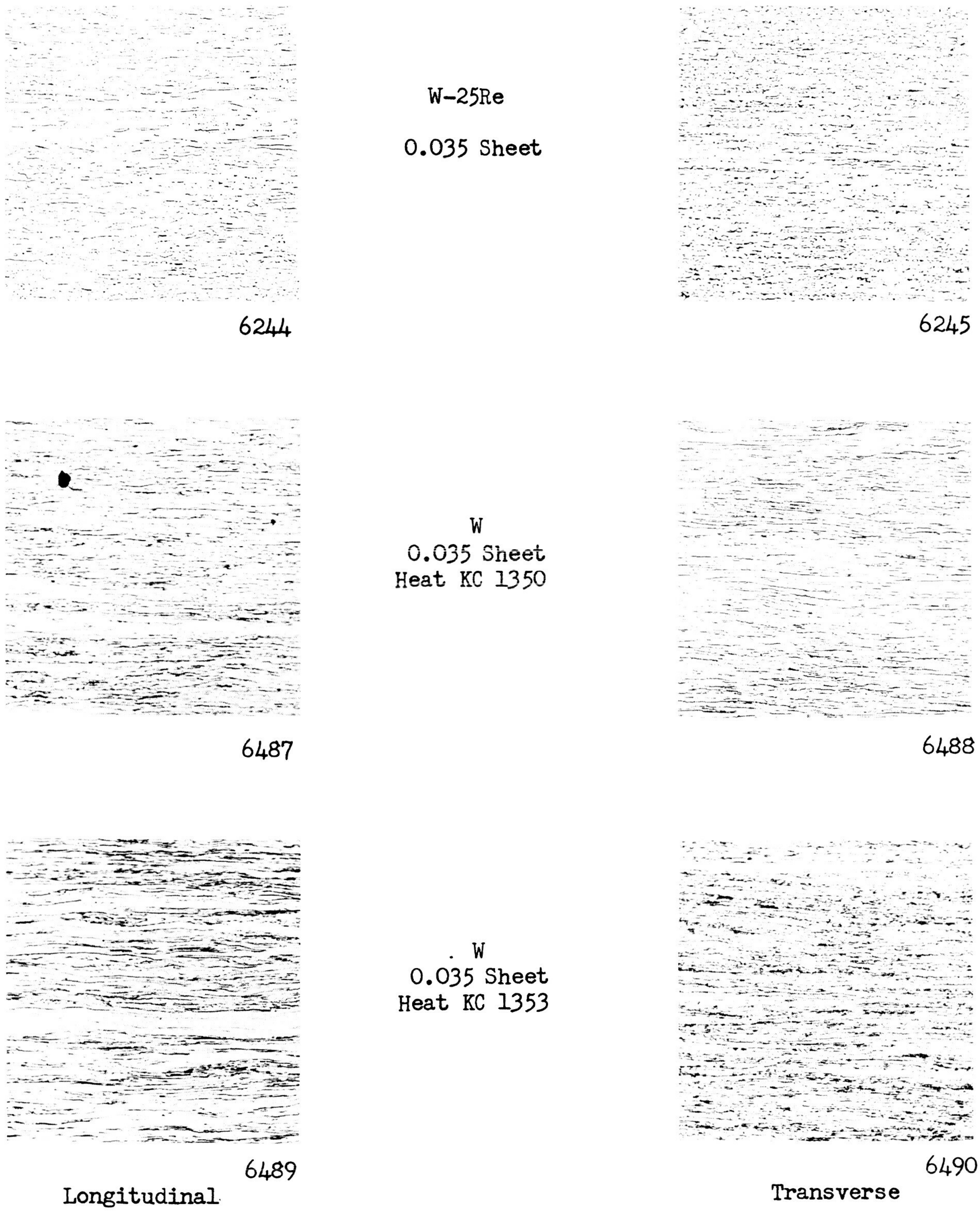


FIGURE 2 - As-Received Microstructure of the Arc Cast W and W-25Re Alloy Sheet

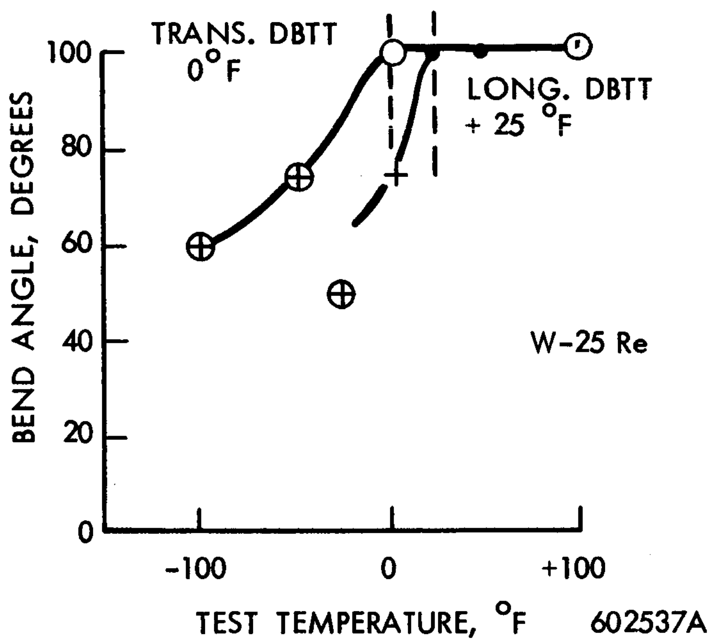


FIGURE 3 - W-25 Re Base Metal Bend Test Results

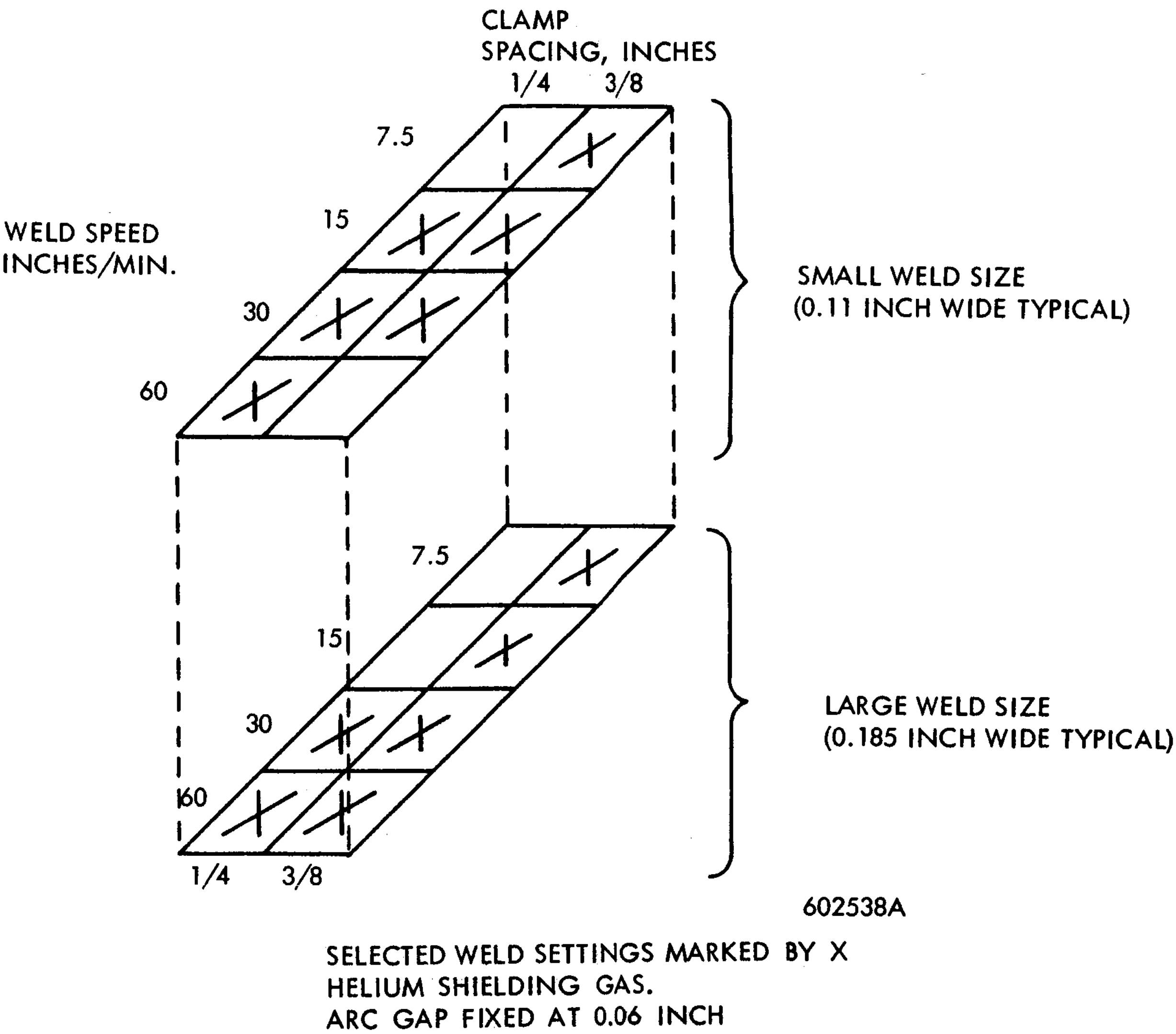


FIGURE 4 - Typical TIG Sheet Butt Weld Schedule

each alloy giving weld size as a function of heat input. A weld programmer was used to vary the current. Hence, only one weld was required per curve. Curves were generated for each alloy at the fifteen inch per minute welding speed using both the 3/8 inch and 1/4 inch clamp spacing. A typical weld, weld data obtained, and type of curve generated using this technique are shown in Figure 5. A set of curves for FS-85 is shown in Figure 6. These curves provided settings for welding at 15 inches per minute and were employed in estimating weld settings at 7.5, 30, and 60 inch per minute weld speeds.

2. Electron Beam Sheet Welding

A complete set of welds were produced for the Cb-752 and SCb-291 alloys in the weld parameter evaluation series. All twenty-four of these welds were bend tested. The bend test results are given in Figures 7 through 10 and the weld parameters are given in Tables 3 and 4. The bend transition temperatures indicated are, as described in the previous quarterly report, those at which a 90° to 105° bend was obtained without cracking on the tension side of the specimen.

3. Restraint Test

The weld restraint test series was continued with the welding of the bead-on-plate patch test for the W-25Re alloy sheet. The second attempt for this test is shown in Figure 11. The first patch test was welded with insufficient penetration and fractured in the heat-affected zone while the second attempt fractured generally along the weld center line. Welding was accomplished without preheating. A need for preheating, however, was indicated by this test and in trial butt welding tests of this alloy.

4. Plate Welding

Following successful trial welding of the six alloys available in plate form, all plate material was laid out, cut to size, and machined with the double "U" weld joint configuration. Machining of these alloys for the entire program has been completed. Welding studies beyond the prototype tests, however, have not yet been initiated. A typical machined and ready to weld plate is shown in Figure 12.

C. EQUIPMENT AND PROCEDURES

1. Titanium Ribbon Purifier

As part of our continuing program to improve the quality of the TIG

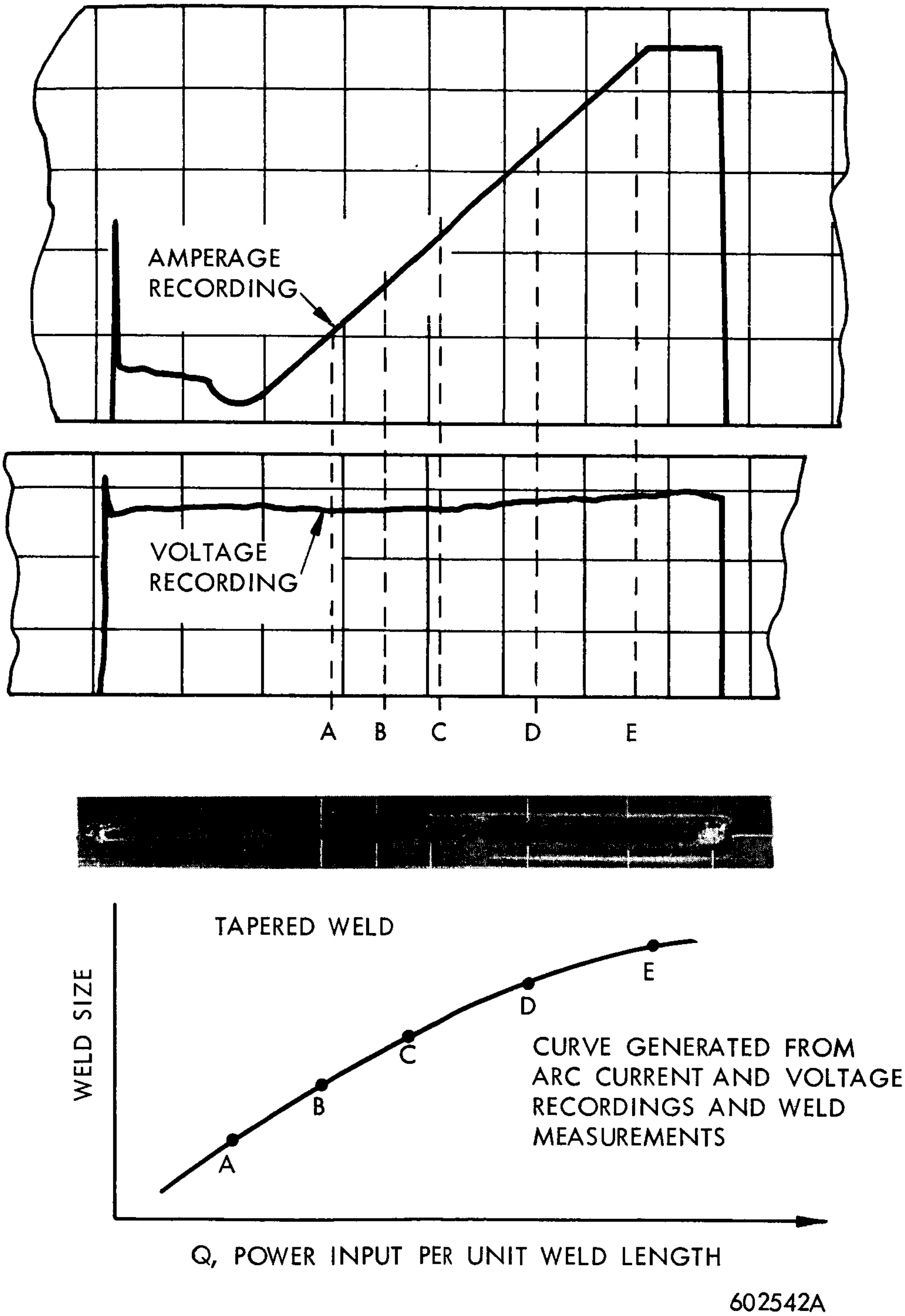


FIGURE 5 - TIG Weld Parameter Test for Weld Size vs. Unit Weld Length Heat Input

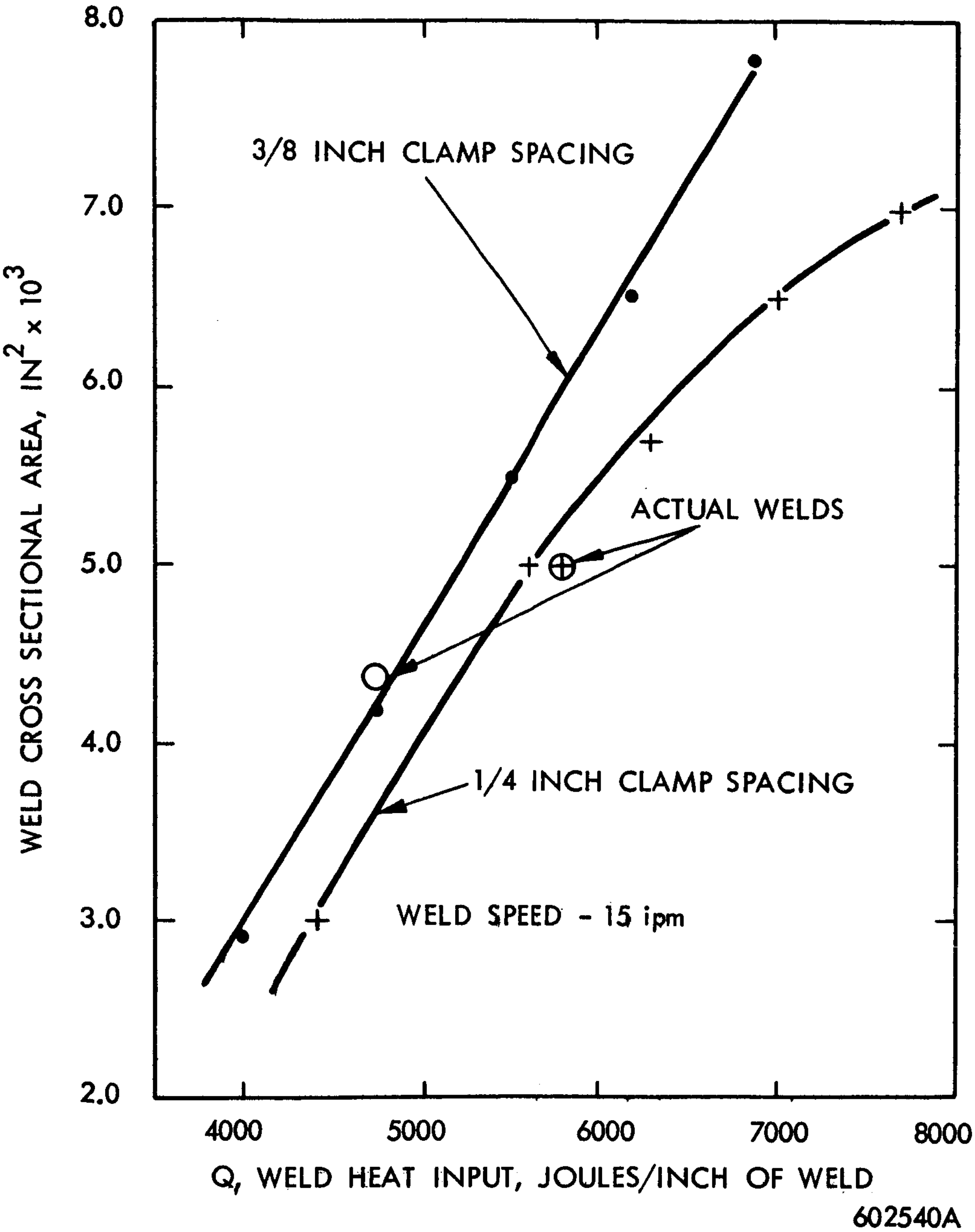


FIGURE 6 - Weld Size As A Function of Heat Input for FS-85

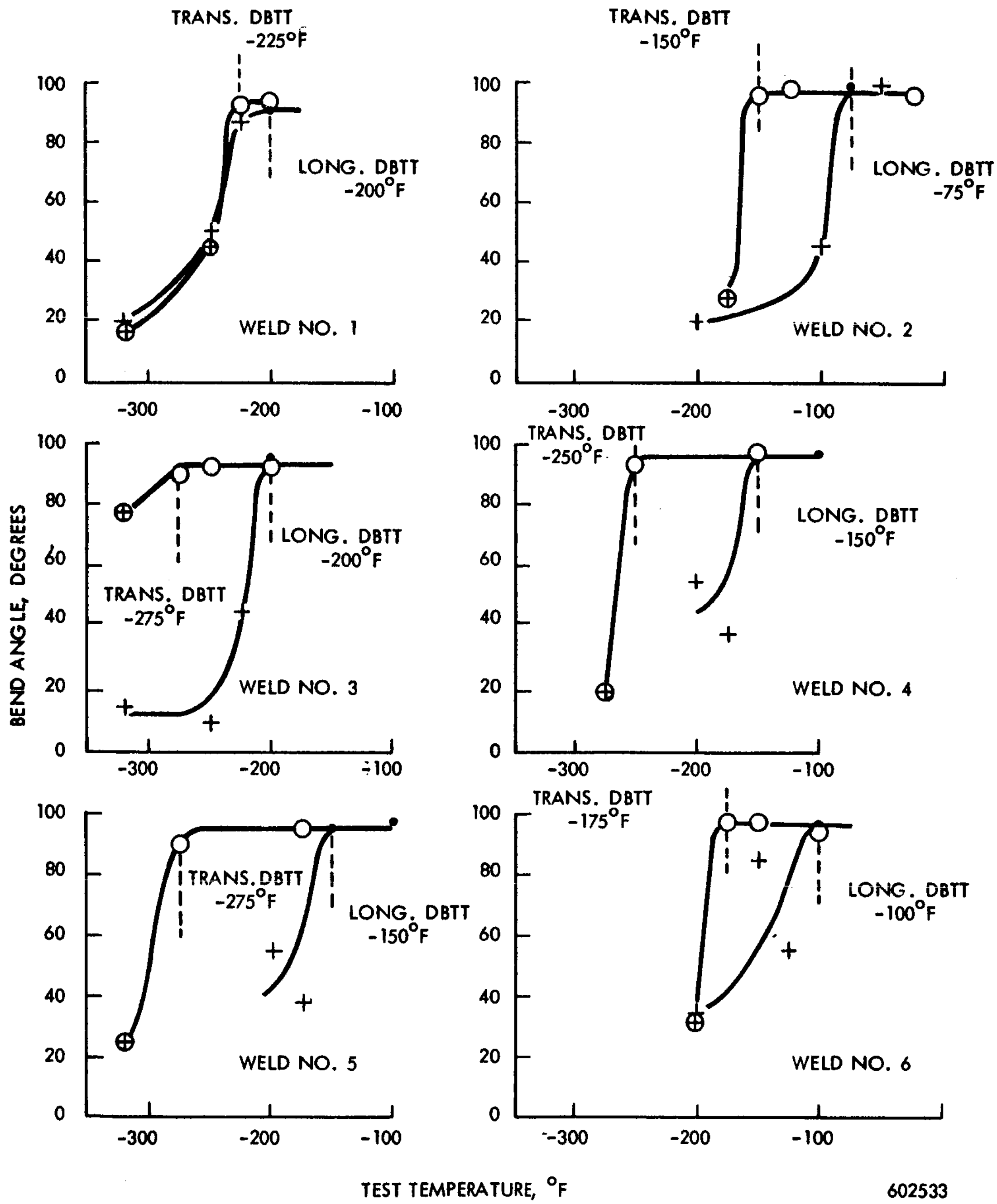


FIGURE 7 - Cb-752 EB Weld Bend Test Results

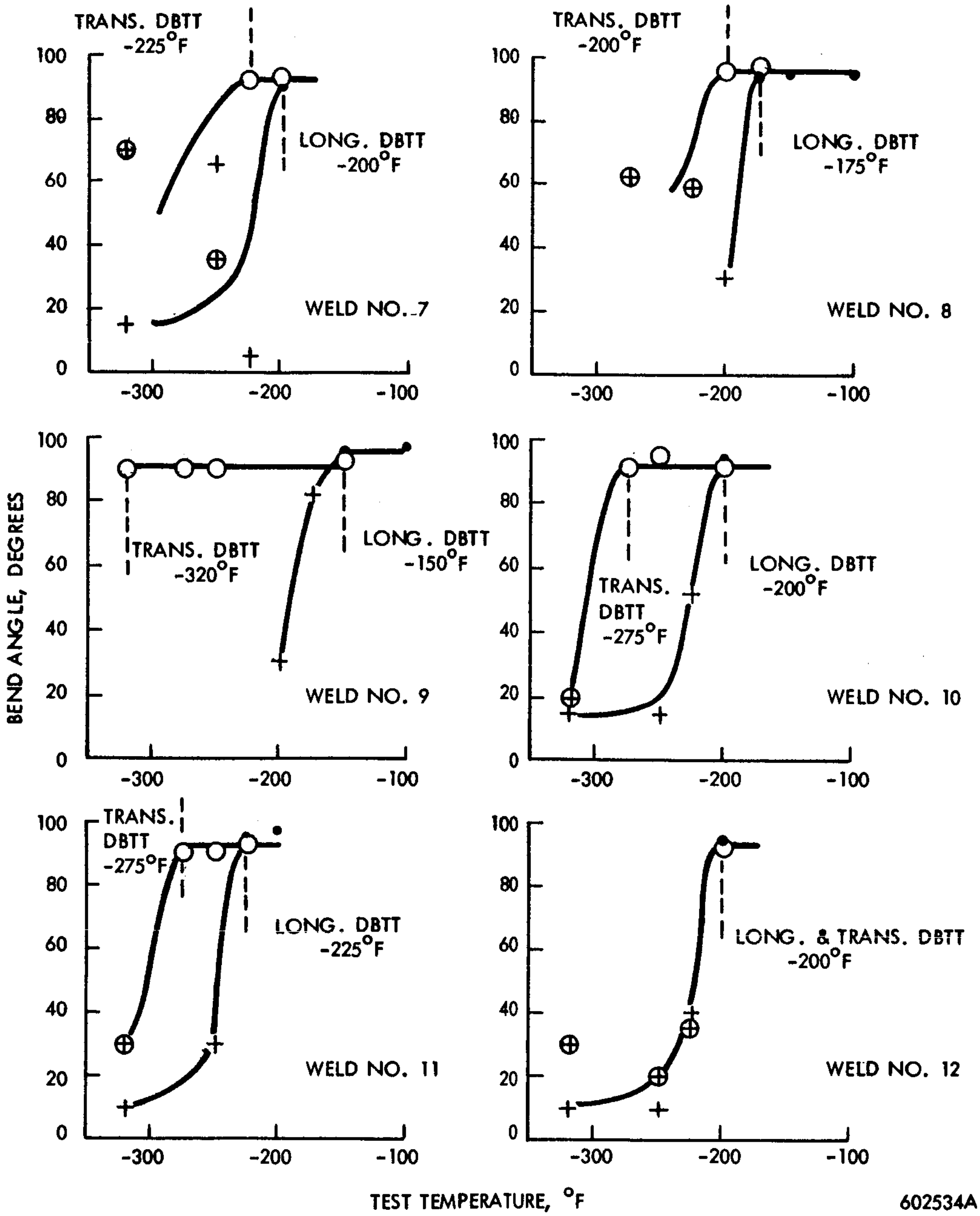


FIGURE 8 - Cb-752 Weld Bend Test Results

TABLE 3 - Electron Beam Welding Parameters for Cb-752

Weld No.	Speed (ipm)	Deflection[1] (inches)	Current (ma)	Chill Spacing (inches)	Power (watts)	Watt-Sec. per inch	Weld Bead Width (inches)		Vacuum[2] (Torr)
							Top	Bottom	
1	100	none	5.0	0.250	750	450	0.025	0.019	6.5×10^{-6}
2	100	L-0.050"	5.0		750	450	0.035	0.022	6.5×10^{-6}
3	50	L-0.050"	4.4		660	790	0.043	0.027	6.5×10^{-6}
4	25	L-0.050"	3.8		570	1370	0.050	0.039	6.0×10^{-6}
5	15	L-0.050"	3.3		500	2000	0.054	0.046	6.0×10^{-6}
6	15	T-0.050"	3.3		500	2000	0.075	0.056	6.0×10^{-6}
7	100	none	5.0	0.094	750	450	0.026	0.018	6.5×10^{-6}
8	100	L-0.050"	5.0		750	450	0.035	0.022	6.5×10^{-6}
9	50	L-0.050"	4.4		660	790	0.042	0.026	6.5×10^{-6}
10	25	L-0.050"	3.8		570	1370	0.045	0.031	6.0×10^{-6}
11	15	L-0.050"	3.3		500	2000	0.036	0.017	6.0×10^{-6}
12	15	T-0.050"	3.3		500	2000	0.054	0.045	6.0×10^{-6}

1. L. is longitudinal
   T. is transverse

2. Current evacuation practice provides pressures of 1.5×10^{-6} Torr

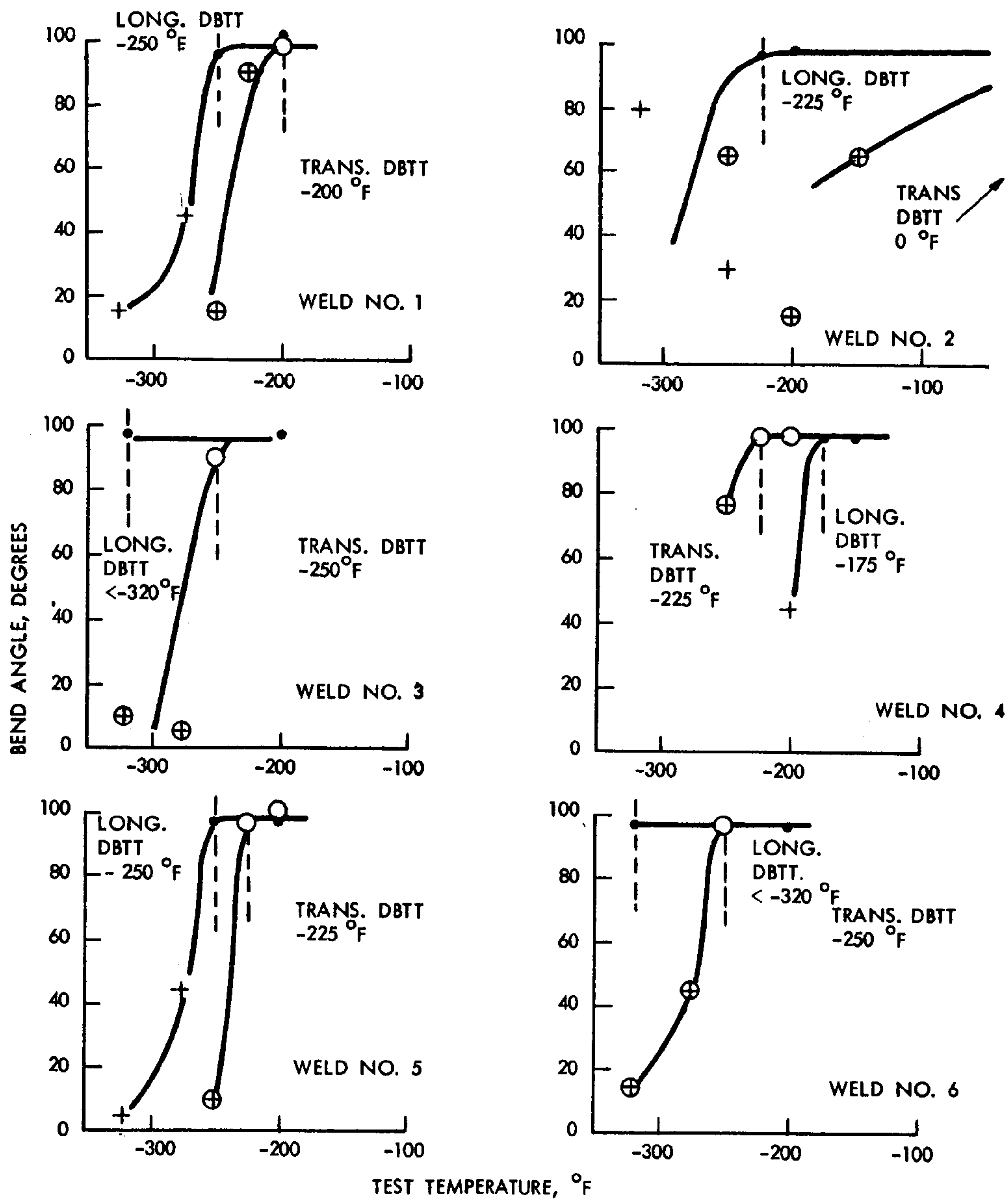


FIGURE 9 - SCb-291 EB Weld Bend Test Results

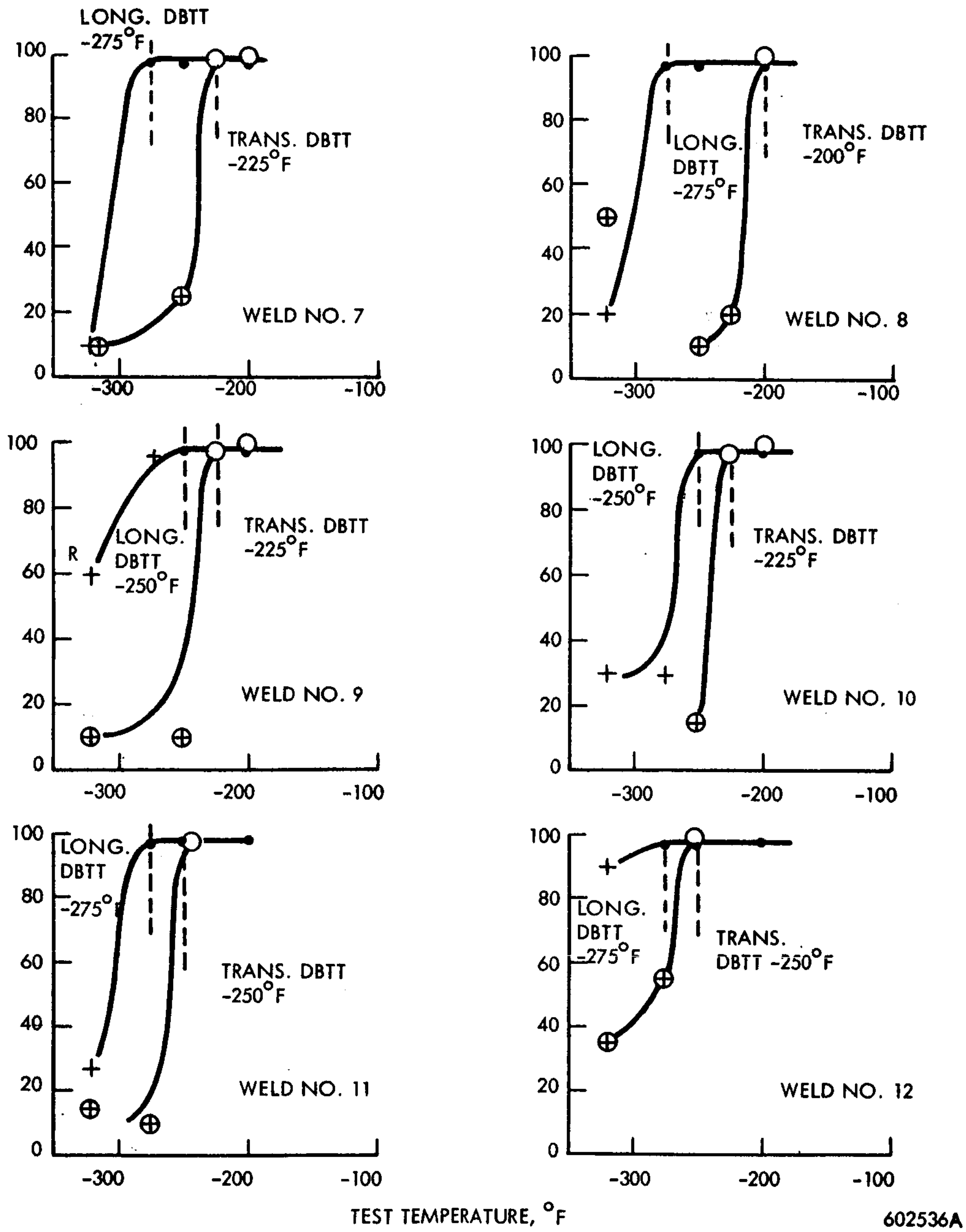


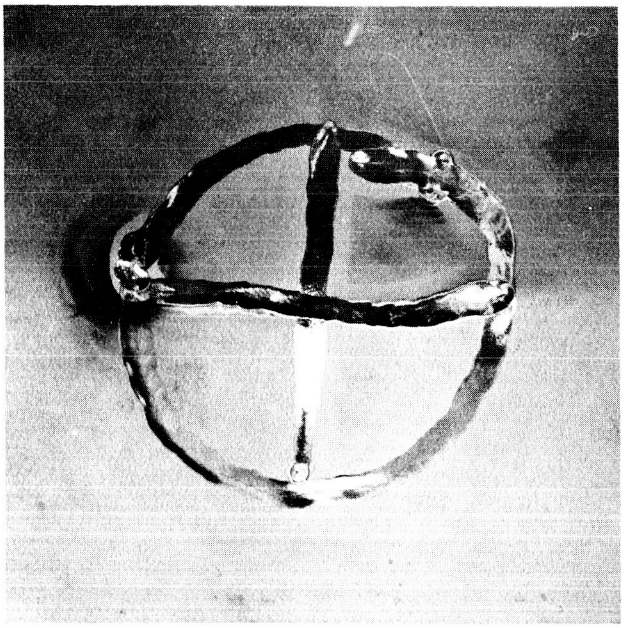
FIGURE 10 - SCb-291 EB Weld Bend Test Results

TABLE 4 - Electron Beam Welding Parameters for SCb-291

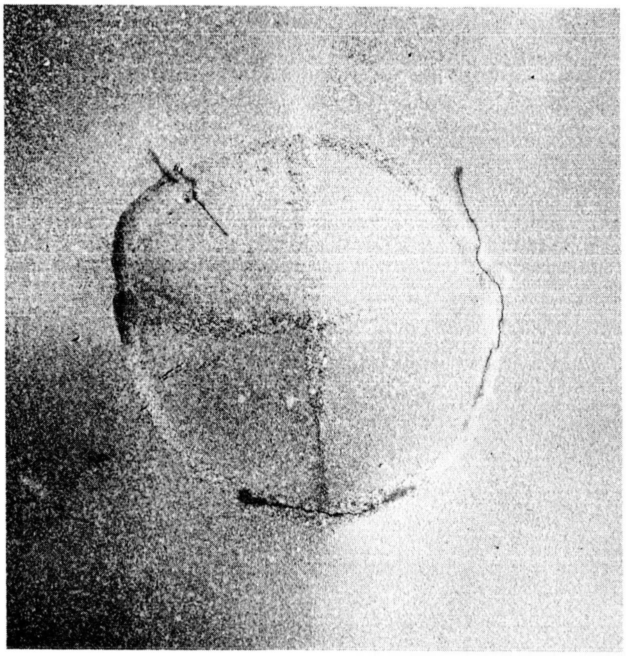
Weld No.	Speed (ipm)	Deflection[1] (inches)	Current (ma)	Chill Spacing (inches)	Power (watts)	Watt-Sec. per inch	Weld Bead Width (inches)		Vacuum[2] (Torr)
							Top	Bottom	
1	100	none	5.0	0.250	750	450	0.028	0.018	4×10^{-6}
2	50	none	4.0		600	720	0.035	0.022	4×10^{-6}
3	50	L-0.050"	4.4		660	790	0.038	0.027	4×10^{-6}
4	25	none	3.1		465	1100	0.038	0.029	5.5×10^{-6}
5	25	T-0.050"	3.9		585	1400	0.070	0.060	5.5×10^{-6}
6	15	none	2.5		375	1500	0.030	0.021	5.5×10^{-6}
7	100	none	5.0	0.094	750	450	0.028	0.016	4×10^{-6}
8	50	none	4.0		600	720	0.033	0.020	4×10^{-6}
9	50	L-0.050"	4.4		660	790	0.040	0.025	4×10^{-6}
10	25	none	3.1		465	1100	0.033	0.024	5.5×10^{-6}
11	25	T-0.050"	3.9		585	1400	0.065	0.050	5.5×10^{-6}
12	15	none	2.5		375	1500	0.023	0.016	5.5×10^{-6}

1. L. is longitudinal
   T. is transverse

2. Current evacuation practice provides pressures of 1.5×10^{-6} Torr



As-Welded (1x)



Dye Penetrant Inspected (1x)

FIGURE 11 - W-25Re Bead-on-Plate Weld Restraint Patch Test

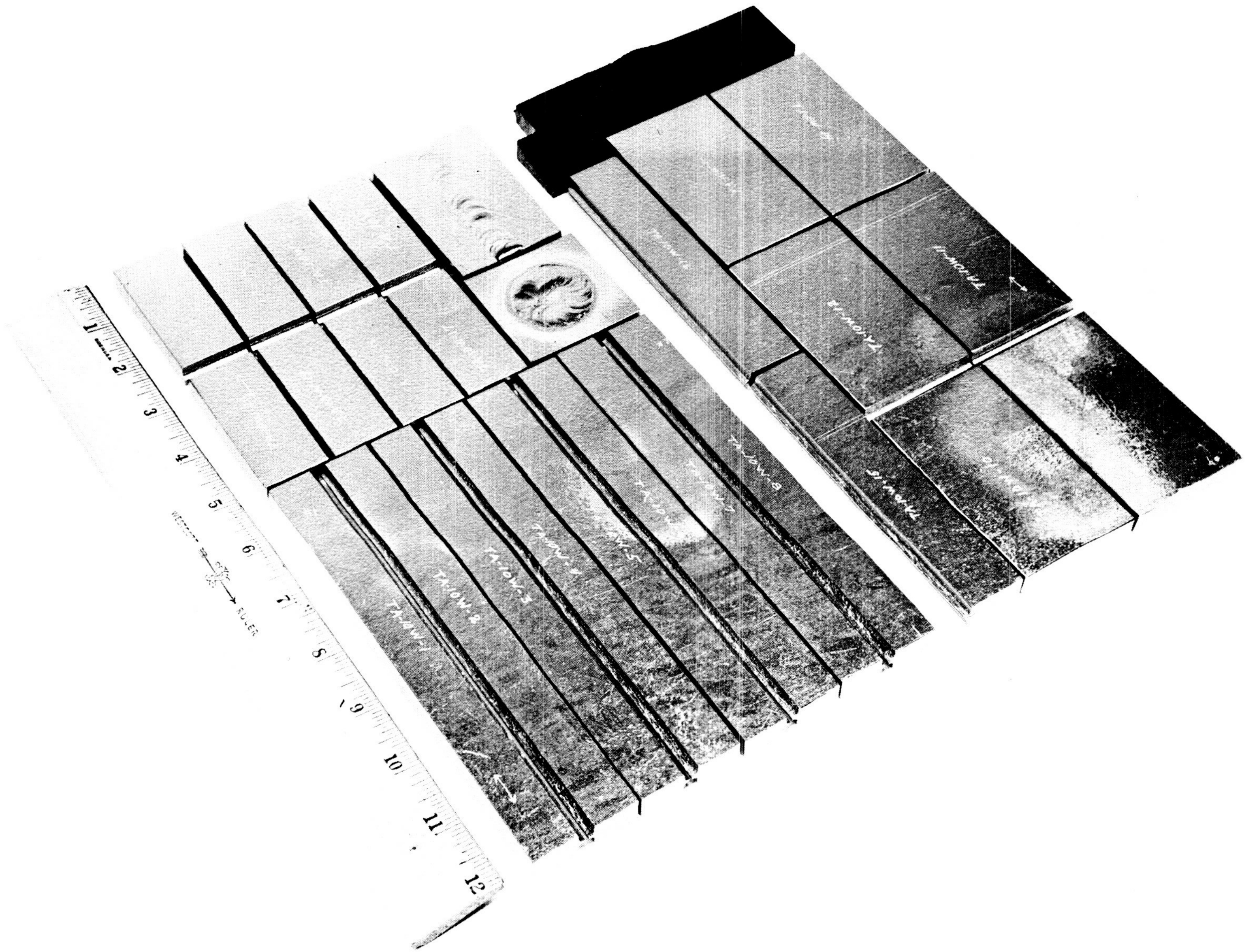


FIGURE 12 - Ta-10W Plate, Machined and Pickled in Preparation for Welding

weld chamber atmosphere, a heated ribbon titanium purifier was evaluated. A successful purifier of this type would avoid the normal complications associated with the lines, valves, pump and fittings of a recirculator purifier. Titanium heated above 1500°F combines with oxygen and nitrogen impurities in the helium atmosphere. Water would also be removed by decomposition, producing hydrogen. In practice a strip of titanium ribbon would remain heated in the chamber to continually purify the atmospheres while welding.

The following experimental procedure was employed for this evaluation. Titanium foil 0.006 inch thick was cut into ribbon samples 36 inches long by 0.030 inches to 0.060 inches wide. The foil was cleaned and pickled and attached to alligator clips. It was necessary to heat and vacuum outgas the foil prior to use as a helium purifier to prevent the evolution of hydrogen into the chamber atmosphere. Hydrogen evolution was noted in an initial test by an abrupt drop in the indicated oxygen impurity content as measured by a Westinghouse O_2 partial pressure gage which is sensitive to a combustible gas background.

A 0.030 inch wide ribbon which had a room temperature resistance of 3.6 ohms was run at 2200°F at 90 volts and approximately 600 watts in a helium atmosphere welding chamber. The chamber was evacuated to 5×10^{-6} and backfilled with Grade A helium. Figure 13 shows the initial impurity content and the effect of ribbon heating. A small reduction in O_2 content was observed and the steady rise in moisture content was halted during operation of the ribbon. Future tests will be run with a much larger quantity of titanium in case the equilibrium solubility of oxygen in titanium is much less than expected at the low partial pressures encountered. Table 5 lists the calculations for the total purification possible for a given titanium size. The 1/2 gram ribbons used should be capable of removing 15 ppm of oxygen from the 60 foot^3 welding chamber. A test with a larger titanium ribbon is contemplated.

A side effect of the ribbon heating was the chamber atmosphere heating that occurred, particularly directly above the ribbon. The use of higher power ribbons would require a heat exchanger to hold the temperature rise to reasonable limits.

2. TIG Welding Fixtures

The sheet butt weld clamp down fixture was modified by replacing the copper clamp inserts with molybdenum inserts. This greatly reduced edge flashing which had been observed at narrow (1/4 inch) clamp spacing.

Since difficulty was encountered in trial welds of the W-25Re sheet, it was decided that weld preheating would be desirable for this alloy and probably necessary for welding unalloyed tungsten and Sylvania "A". A modified backup

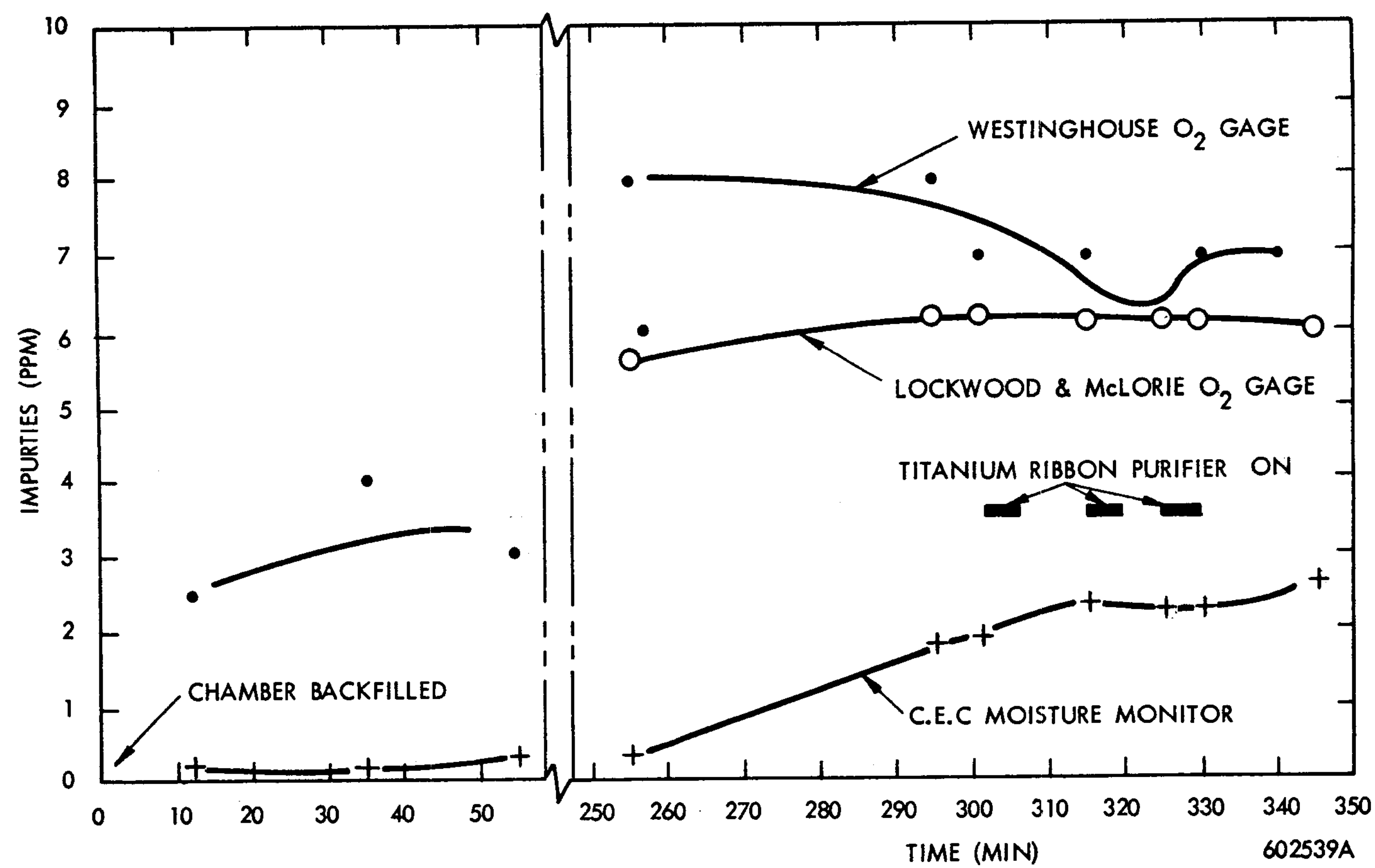


FIGURE 13 - Effect of Titanium Ribbon Purifier on Weld Chamber Atmosphere

TABLE 5 - Calculations for Titanium Ribbon Gettering Capacity

I. Total weight of O_2 present in 60 ft.^{3} welding chamber as a function of ppm.

(1 ppm) = 1 x 10^{-6} (volume fraction)

(2000 liters) x (1 x 10^{-6}) x (1.42 gm O_2/liter) = 2.8 mg O_2 per ppm O_2 in chamber.

II. Total weight of O_2 soluble in titanium ribbon.

0.500 gm ribbon x 10 w/o O_2 solubility = 0.0500 gm O_2 = 50 mg O_2

III. Gettering Capacity of Ribbon

50 mg O_2/2.8 mg O_2 per ppm O_2 in chamber = 18 ppm O_2

Assumed to be reasonable from data of Wasilewski and Kehl, Ref. No. 1, although equilibrium at low partial pressure of O_2 may be much lower.

bar was designed and fabricated for use in the welding fixture. It has an integral heater and is also equipped with a cooling channel to reduce weld cycle time should this become desirable.

IV. FUTURE WORK

Weld parameter optimization studies will be completed for TIG butt welded sheet for seven of the available alloys, and for EB butt welded sheet for two additional alloys, W-25Re and D-43.

Plate butt welding studies and bend testing will be initiated.

An additional weld box atmosphere decay test will be run using latex natural rubber gloves. This will compliment previous tests run using neoprene, butyl rubber, and polyvinyl chloride gloves.

The high vacuum annealing furnaces performance will be checked using a residual gas analyzer to determine the nature of the gas load during bakeout, furnace start-up, and furnace operation. In this test a titanium sublimation pump will be used to provide supplemental pumping capacity. The residual gas analyzer should also prove useful in evaluating the sublimation pump performance.

V. REFERENCES

1. R. J. Wasilewski and G. L. Kehl, "Diffusion of Nitrogen and Oxygen in Titanium", Journal of Institute of Metals, 1954-1955, Volume 83.